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following sample preparation. This is applicable to organic and inorganic chemical analyses.

3.7 Preparation Batch: A group of 20 or less client samples processed together under the same conditions, within an 8 hour working shift.

3.8 Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ is also referred to as the method quantitation limit (MQL) or the reporting limit (RL).

3.9 Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent.

3.10 Method Detection Limit (MDL) study: the procedure, as described in 40CFR part 136, for determining the LOD based on statistical analysis of 7 low-level replicate spikes.

4) Health and Safety Warnings

4.1 Lab Safety: Due to various hazards in the laboratory, safety glasses and laboratory coats or aprons must be worn at all times while in the laboratory. In addition, gloves and a face shield should be worn when dealing with toxic, caustic, and/or flammable chemicals.

4.2 Chemical Hygiene: The toxicity or carcinogenicity of each reagent used has not been precisely defined; however, each chemical used should be treated as a potential health hazard. Exposure to laboratory reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.3 Waste Management: The principal wastes generated by this procedure are the method-required chemicals and standards. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required. Laboratory procedures in SOP HN-SAF-001, Waste Disposal Procedures, must be followed.

4.4 Pollution Prevention: The materials used in this method pose little threat to the environment when recycled and managed properly. The quantities of chemicals purchased should be based on the expected usage during its shelf life. Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 Mercury is a known health hazard and can be readily absorbed via the respiratory tract in both elemental vapor and mercury compound dusts. All work must be completed under proper ventilation, utilizing appropriate personal protective equipment.

5.2 This procedure utilizes strong acids and oxidizers. Appropriate personal protective equipment shall be used.

6) Interferences

6.1 Potassium permanganate is added to eliminate possible interference from sulfide.

6.2 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities - This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst - It is the responsibility of the analyst(s) to:

7.2.1 Each must read and understand this SOP and follow it as written. Any deviations or non-conformances must be documented and submitted to the QA Manager for approval.
7.2.2 Produce method compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).
7.2.3 Complete the required initial demonstration of proficiency before performing this procedure without supervision.
7.2.4 Complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
7.2.5 The analysts must submit data for peer or supervisor review.

7.3 Section Supervisor - It is the responsibility of the section supervisor to:

7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.
7.3.2 Ensure analysts have completed the required initial demonstration of proficiency before performing this procedure without supervision.
7.3.3 Ensure analysts complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
7.3.4 Ensure analysts produce method compliant data that meet all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

7.4 Project Manager - It is the responsibility of the Project Manager to ensure that all method requirements for a client requesting this procedure are understood by the laboratory prior to initiating this procedure for a given set of samples.

7.5 QA Manager: The QA Manager is responsible for

7.5.1 Approving deviations and non-conformances
7.5.2 Ensuring that this procedure is compliant with method and regulatory requirements,
7.5.3 Ensuring that the analytical method and SOP are followed as written through internal method and system audits.

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8) Sample Collection, Handling, and Preservation

8.1 Solid samples are collected in 4 oz. glass containers and stored at 4°C.

8.2 The holding time is 28 days

9) Equipment and Supplies

9.1 Cetac M-6100A Mercury Analyzer.

9.2 Analytical balance - Satorius ED124S (or equivalent)

9.3 Hotblock (or hotplate) unit, capable of maintaining a temperature of 90-95°C.

9.4 Adjustable pipets, 0.1 - 1.0 ml and 0.5 - 5.0 ml

9.5 70 ml digestion cups, verified for volume.

9.6 Teflon chips (mercury free)

10) Standards and Reagents

10.1 ASTM Type II water (DI).

10.2 Sulfuric acid (H_2SO_4), concentrated (36N), ACS grade.

10.3 Hydrochloric acid (HCl), concentrated, Trace Metals grade

10.4 Nitric acid (HNO_3), concentrated, Trace Metals grade

10.5 Stannous chloride, reagent grade or higher

10.6 Stannous chloride, 10 %

10.6.1 Add 100g stannous chloride and 50ml concentrated H_2SO_4 to approximately 300ml DI water

10.6.2 Bring to a final volume of 1000ml with DI water.

10.6.3 This solution should be clear, not cloudy or yellow. If cloudiness or yellowing occur, re-prepare

10.6.4 Solution is good for 1 week if kept from reacting with open air.

10.7 Sodium chloride, reagent grade or higher

10.8 Hydroxylamine hydrochloride, reagent grade or higher

10.9 Sodium chloride-hydroxylamine hydrochloride solution:

10.9.1 Dissolve 120g of sodium chloride and 120g of hydroxylamine hydrochloride in approximately 700ml of DI water.

10.9.2 Bring to a final volume of 1000ml.

10.10 Potassium permanganate - mercury analysis grade

10.11 Potassium permanganate 5% solution (w/v)

10.11.1 Dissolve 125 g of mercury free potassium permanganate in approximately 700ml of DI water

10.11.2 Bring to a final volume of 2500ml with DI water

10.11.3 Ample stirring will be required to solubilize the KMnO4

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10.12 Potassium persulfate, reagent grade or higher

10.13 Potassium persulfate 5% solution (w/v)

10.13.1 Dissolve 50g potassium persulfate in approximately 500ml of DI water (heating and stirring may be needed to aid in dissolution.)
10.13.2 Bring to a final volume of 1000ml with DI water

10.14 Aqua Regia
10.14.1 Prepare aqua regia fresh before use.
10.14.2 Carefully add 1 part Nitric Acid (10.4) to 3 parts Hydrochloric Acid (10.3)
10.14.3 Estimate the amount needed to be prepared based on the number of samples to be processed. Always maintain the 1:3 HNO3:HCl ratio.
10.14.4 Discard any portion of aqua regia not utilized via the acid waste stream.

10.15 Stock Mercury Calibration Standard, 1000 ug/ml- purchase as certified standard

10.16 Intermediate Hg Calibration Standard, 10.0 ug/ml

10.16.1 Pipet 1.0 ml of 1000 ug/ml Stock Mercury Standard into a 100ml volumetric flask containing ~ 50ml acidified DI water and dilute to volume.
10.16.2 Transfer to a clean appropriately labeled container.
10.16.3 Prepare every six months or if degradation is noted. The expiration date must not exceed that of the parent stock.

10.17 Working Hg Calibration Standard, 0.1 ug/ml

10.17.1 Pipet 1.0 ml of 10.0 ug/ml Intermediate Hg Cal Standard (10.16) into a 100 ml volumetric flask containing ~ 50ml acidified DI water
10.17.2 Bring to volume.
10.17.3 Prepare fresh weekly.

10.18 Stock Mercury Calibration Verification Standard, 1000 ug/ml - purchase as certified standard, from a second source supplier

10.18.1 Working Hg Calibration Verification Standard @ 0.1ug/ml: Using the stock second source standard, follow the procedures established in Section 10.16 and 10.17.

10.19 Mercury Initial Calibration Verification Standard (ICV) @ 2.0ug/L:

10.19.1 Prepare the under the same conditions as the 2.0ug/L calibration point in Table 11.1 utilizing the the Working Calibration Verification Standard (10.18.1).

10.20 Mercury Continuing Calibration Verification Standard (also called Instrument Performance Check or IPC) @ 2.0 ug/L:

10.20.1 Dilute 1 ml of the Working Calibration Standard (10.17) to 50 ml with DI water.

10.21 Mercury Low-Level Continuing Calibration Verification Standard (CRDL) @ 0.2 ug/L

10.21.1 Dilute 100 uL of the Working Calibration Standard (10.17) to 50 ml with DI water.

10.22 Mercury LCS @ 2.0 ug/L:

10.22.1 Place ~0.600g Teflon chips in a 70 ml digestion cup along with 5ml DI water, and spike with 1.0 ml of the 0.1 ug/ml Working Calibration Standard (Section 10.17).

10.23 Mercury MS/MSD @ 2.0 ug/L:

10.23.1 Place ~0.600g of sample in a 70 ml digestion cup along and spike with 1.0 ml of the 0.1 ug/ml Working Verification Standard (Section 10.18.1).

10.23.2 Spiking must occur prior to the addition of reagents.

11) Method Calibration

11.1 Mercury Calibration Curve Standards, 0 - 4.0 ug/L: Volume additions of the standards are made using volume calibration verified pipets.

Table 11.1: Calibration Curve Preparation

Calibration Curve Levels, ug/L Hg	**0**	**0.2**	**0.5**	**1.0**	**2.0**	**4.0**
ml of 0.1 ug/ml Hg added (10.17)	0	0.10	0.25	0.50	1.0	2.0
ml of Sulfuric Acid (10.2)	2.5	2.5	2.5	2.5	2.5	2.5
ml of Nitric Acid (10.4)	1.25	1.25	1.25	1.25	1.25	1.25
ml of 5% KMnO4 (10.11)	7.5	7.5	7.5	7.5	7.5	7.5
ml of 5% K2S2O8 (10.13)	4.0	4.0	4.0	4.0	4.0	4.0
ml of Hydroxylamine-HCl/NaCl (10.9)	3.0	3.0	3.0	3.0	3.0	3.0
Final Vol w/DI (ml)	50	50	50	50	50	50

Note: The analyst may utilize additional standards as needed to extend the calibration range.

11.2 The calibration curve is digested on a daily basis under conditions equivalent to those used for the preparation of samples.

11.3 CETAC M6100A:

11.3.1 Must use the Linear Regression mode of analysis for establishment of the initial calibration

11.3.2 Instrument response is an average of a minimum of 3 replicates, and must have a %RSD <5% for concentrations greater than or equal to the lowest calibration point.

11.3.3 Will plot a calibration curve and determine the slope, intercept and the correlation coefficient, r.

11.3.4 The curve must generate an "r" of 0.995 or better for acceptance.

11.3.5 The stored calibration curve information will calculate the mercury concentration in all samples analyzed after the acceptance of the curve.

11.4 An ICV standard (10.19) must be analyzed with each new calibration. Acceptance criteria are 90-110% of the spiked concentration.

11.5 An IPC/CCV standard (10.20) must be analyzed with each new calibration. The IPC

must be analyzed immediately following the ICB with acceptance criteria of 95-105% of the spiked concentration. The CCV must then be read after each 10 subsequent samples and at the end of the sequence with acceptance criteria of 90-110% of the spiked concentration.

11.6 A CRDL standard (10.21) must be analyzed immediately following the IPC/CCV standard. Acceptance criteria are 60-140% of the spiked concentration. This standard is carried through the digestion process, daily, when client samples are to be analyzed.

11.7 A Continuing calibration blank (CCB) must be analyzed immediately following the CRDL standard. This blank is used to demonstrate instrument cleanliness. Refer to section 16.5.5 for acceptance criteria.

12) Sample Preparation/Analysis

12.1 Sample Preparation:

12.1.1 Transfer approximately 0.600g of a thoroughly mixed representative sample (Teflon chips for MB and LCS) to a 70 ml digestion cup.
12.1.2 Add 5ml DI H_2O and 5.0ml of aqua regia
12.1.3 Place samples on the hotblock and heat for 2 minutes. Remove and allow to cool.
12.1.4 Add 7.5ml of potassium permanganate.

12.1.4.1 The purple potassium permanganate solution color must persist for at least 15 minutes.
12.1.4.2 If, not, add an additional 7.5 ml of potassium permanganate.
12.1.4.3 If a sample still consumes the potassium permanganate, discard the sample and perform necessary dilutions.

12.1.5 Heat for 30 minutes, at 95 +/- 3°C. Record digestion temperature.
12.1.6 Place a ribbed watch glass on the samples during digestion.
12.1.7 Remove cups from digestion unit and cool to room temperature.
12.1.8 Add 3 ml hydroxylamine hydrochloride solution. Mix to clear the permanganate color.
12.1.9 Bring to a final volume of 50ml with DI.
12.1.10 Proceed to Section 12.2.

12.2 Analysis

12.2.1 Allow particulates to settle or filter the digestate through a 0.45um filter.
12.2.2 Transfer about 15 ml of each digestate to an autosampler vial.
12.2.3 Note: The instrument performs the addition of the stannous chloride solution during the automated run.
12.2.4 Create a sequence table with the CETAC M6100A ALS template and start sequence.
12.2.5 Typical Analytical Sequence:

12.2.5.1 5-Point Initial Calibration curve and a blank
12.2.5.2 Initial Calibration Verification standard
12.2.5.3 Initial Calibration Blank
12.2.5.4 IPC/CCV
12.2.5.5 CRDL
12.2.5.6 Continuing Calibration Blank
12.2.5.7 Method Blank
12.2.5.8 Laboratory Control Sample
12.2.5.9 Commercial sample
12.2.5.10 Matrix spike
12.2.5.11 Matrix spike duplicate
12.2.5.12 Commercial samples (5)
12.2.5.13 IPC/CCV
12.2.5.14 CRDL
12.2.5.15 Continuing Calibration Blank
12.2.5.16 Additional samples or QC (10)
12.2.5.17 IPC/CCV
12.2.5.18 CRDL
12.2.5.19 Continuing Calibration Blank

13) Troubleshooting

13.1 Refer to CETAC M6100A Hardware manual for specific troubleshooting guidance.

14) Data Acquisition

14.1 Create a prep batch in LIMS. This information will apply to the final calculations.

14.2 Concentration data from the Cetac M6100A is downloaded directly into the data entry section of LIMS.

14.3 Dilutions must be entered manually into the LIMS system under the appropriate field (DF).

15) Calculation, and Data Reduction Requirements

15.1 QC Calculations: LIMS calculates the percent recovery for various QC samples (MS, MSD, LCS) according to the following equations:

15.1.1 % Recovery, %R (for MS and MSD Samples)

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where:
SSR = Spiked Sample Result (mg/L or mg/kg).
SR = Sample Result (unspiked).
SA = Spike Amount Added (mg/L or mg/kg).

15.1.2 % Recovery, %R (for standards and LCS)

$$\%R = \frac{(SSR)}{SA} \times 100$$

Where:
SSR = Spiked Sample Result (mg/L or mg/kg).
SA = Spike Amount Added (mg/L or mg/kg).

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15.1.3 RPD (for precision or duplicate evaluation)

$$RPD = \frac{|SR_1 - SR_2|}{½(SR_1 + SR_2)} \times 100$$

Where:
SR_1 = Sample result for duplicate 1.
SR_2 = Sample result for duplicate 2.

16) Quality Control, Acceptance Criteria and Corrective Action

16.1 Initial Calibration Curve

16.1.1 Utilize 5 standards (minimally) and a blank
16.1.2 Instrument response is an average of a minimum of 3 replicates, and must have a %RSD <5% for concentrations greater than or equal to the lowest calibration point.
16.1.3 Acceptance Criteria: "r" must be 0.995 or better.
16.1.4 Must be established daily at a minimum or as required by CCV acceptance failure
16.1.5 ICal Curve Failure Corrective Action:

16.1.5.1 Examine standards and reprocess curve.
16.1.5.2 If curve continues to fail, check support equipment and review instrument maintenance logbooks for possible instrument problems. Prepare new standards as necessary and reprocess curve.
16.1.5.3 All samples associated with a failed initial calibration curve must be reprocessed.

16.2 Initial Calibration Verification (ICV)

16.2.1 Utilize a 2.0 ppb standard prepared from a second source supplier
16.2.2 Perform after each initial calibration.
16.2.3 Must meet accuracy criteria of 90-110% of spiked concentration
16.2.4 ICV Failure Corrective Action:

16.2.4.1 Examine standards and reprocess curve/verification standard.
16.2.4.2 If curve continues to fail, check support equipment calibration checks and remake standards.
16.2.4.3 All samples associated with a failed ICV must be reprocessed.

16.3 Instrument Performance Check (IPC) or Continuing Calibration Verification (CCV)

16.3.1 Prepare from the same source as the associated initial calibration standards, whenever a new curve is prepared and as needed thereafter.
16.3.2 Utilize a 2.0 ppb standard prepared from the primary source
16.3.3 Analyze after the initial calibration, prior to sample analysis, every ten samples thereafter, and at the end of the run.
16.3.4 Acceptance Criteria

16.3.4.1 The IPC must be analyzed immediately following the ICB with acceptance criteria of 95-105% of the spiked concentration.

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16.3.4.2 The CCV must then be read after each 10 subsequent samples and at the end of the sequence with acceptance criteria of 90-110% of the spiked concentration.

16.3.5 CCV Failure Corrective Action:

16.3.5.1 Examine standard and reprocess IPC-CCV.
16.3.5.2 If curve continues to fail, check support equipment and process a new initial calibration.
16.3.5.3 All samples associated with a failed IPC-CCV (preceding and following) must be re-analyzed.
16.3.5.4 If the IPC-CCV fails high and all associated samples (preceding and following) are non-detect, sample results may be reported if appropriately narrated as such.

16.4 Low-level Continuing Calibration Verification (MRL/CRDL)

16.4.1 Prepare daily from same source as associated initial calibration standards
16.4.2 Must be processed utilizing the entire digestion procedure
16.4.3 Utilize a 0.2 ppb standard prepared from the primary source
16.4.4 Analyze after the initial calibration, prior to sample analysis, every ten samples thereafter, and at the end of the run.
16.4.5 Acceptance Criteria

16.4.5.1 Must meet accuracy criteria of 60-140% the spiked concentration

16.4.6 LLCCV Failure Corrective Action:

16.4.6.1 Examine standard and reprocess CRDL.
16.4.6.2 If verification continues to fail, check support equipment and process a new initial calibration.
16.4.6.3 All samples associated with a failed CRDL (preceding and following) shall be re-analyzed.
16.4.6.4 If the CRDL fails high and associated samples (preceding or following) are non-detect, sample results may be reported if appropriately narrated as such.

16.5 Blanks (ICB/CCB/MB):

16.5.1 Prepare daily
16.5.2 Method blanks must be processed utilizing the entire digestion procedure.
16.5.3 One blank may serve as the ICB and CCB. If a blank is used interchangeably, it must be designated appropriately (i.e., ICB-CCB) and it cannot exceed the most restrictive acceptance criteria.

16.5.4 Frequency:

16.5.4.1 ICB, CCB, and MBs must be processed as noted in the analytical sequence
16.5.4.2 One method blank per every batch of 20 or less samples must be processed.

16.5.5 Acceptance Criteria:

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16.5.5.1 Mercury concentration should be less than the MDL and must be less than ½ the PQL.

16.5.6 Corrective Action:

16.5.6.1 If ICB/CCB contamination is indicated, reprocess the blank and examine for contamination.
16.5.6.2 If the blank continues to fail, terminate analysis and correct the problem before proceeding. An acceptable blank must be demonstrated before sample analysis resumes.
16.5.6.3 If blank contamination is indicated, all associated samples (before and after) must be reanalyzed.
16.5.6.4 If MB contamination is indicated, reanalyze the blank and examine for contamination.
16.5.6.5 If the MB continues to fail, re-digest all associated samples.
16.5.6.6 If MB contamination is indicated and samples cannot be re-digested, all associated must be reported with a "B" qualifier.
16.5.6.7 A nonconformance report (NCR) must be issued for any sample reporting associated with blank contamination.
16.5.6.8 If a reported mercury result is more than 10 times the blank mercury level, the B flag is not applied to the sample result.
16.5.6.9 If the sample associated with a contaminated MB is "ND", the sample may be reported if appropriately qualified.

16.6 Laboratory Control Sample (LCS):

16.6.1 Prepare from the initial calibration working standard at a theoretical concentration of 2.0 ppb.
16.6.2 Process one LCS per batch of 20 or less sample digestions.
16.6.3 Must meet accuracy performance criteria for outlined in the applicable LIMS test code.
16.6.4 LCS Failure Corrective Action:

16.6.4.1 If the LCS does not meet acceptance criteria, all associated samples in the analytical batch must be re-digested/reanalyzed.
16.6.4.2 If the LCS fails high and associated samples are "ND", they may be reported if appropriately narrated.

16.7 Matrix Spikes (MS/MSD):

16.7.1 Prepare from the initial calibration verification standard at a theoretical value of 2 ppb
16.7.2 Prepare MS/MSD at a 5% frequency for each digestion batch of 20 or less samples.
16.7.3 MS/MSD Recovery Criteria:

16.7.3.1 Must meet accuracy performance criteria as outlined in the applicable LIMS test code.
16.7.3.2 If the spike results or the spike duplicate results are outside the acceptance limits, evaluate against the LCS results to determine whether a matrix effect is present or if laboratory error is suspected. (The LCS must fall within acceptance criteria in order for the data to be accepted.)

16.7.3.3 If laboratory error is suspected, all associated samples must be re-digested/reanalyzed. If samples cannot be re-processed, the associated data must be qualified and narrated as to potential bias.

16.8 Sample Duplicate or Matrix Spike Duplicate

16.8.1 Unless specified otherwise by project requirements, a matrix spike duplicate (MSD) may be processed in place of a sample duplicate.

16.8.2 Duplicates must be analyzed on a frequency of one per 20 or less for SW-846 7471A/B.

16.8.3 If insufficient sample is available for a duplicate analysis, a duplicate on a sample analyzed in a previous batch may be performed.

16.8.4 Acceptance Criteria:

16.8.4.1 Must meet precision performance criteria outlined in the applicable LIMS test code.

16.8.5 Duplicate Failure Corrective Action:

16.8.5.1 If the duplicate analysis does not meet acceptance criteria, the analytical process must be evaluated for possible errors. All samples associated with failed duplicate analyses should be re-processed. If re-processing of the samples cannot be completed, all associated results must be qualified and narrated as to potential bias.

16.9 Deviations and non-conforming events must be documented using a Nonconformance Corrective Action Report (NCAR) or as an Exception Report item on the laboratory review checklist. For mandatory QC failures (e.g. LCS), the NCAR must be submitted to the QA Manager via the NCAR database.

17) Data Records Management

17.1 All data is stored both electronically and hard copy for 10 years.

17.2 All analytical sequence IDs and standard preparation information must be recorded in the Run logbook. Hardcopy computer printouts of analytical sequences and raw data must be retained and initialed by the analyst (electronic initials are acceptable). To simplify standard tracking, analyst must attempt to use one lot of reagents and standards with each batch.

17.3 Complete all pertinent sections in the respective logbooks. If not-applicable then line out the section. "Z" out or "X" out all large sections of the worksheet that are not used. Make all corrections with single line through, date and initial. Make NO obliterations when manually recording data.

17.4 Logbooks are controlled. Never remove a page from a logbook. Completed logbooks are returned to the QA department when filled and no longer needed in the work area.

17.5 The effective date of this SOP is the date in the header or last signature date, whichever is most recent

18) Contingencies for Handling Out of Control Data

18.1 When method required QC exceedances occur, in every case where sample data quality are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.

18.2 When affected sample analysis cannot be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).

18.3 All analysts must report sufficient comments in laboratory data review checklist for exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.

18.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS or using the Laboratory Data review checklist.

18.5 Logbooks must be reviewed monthly by the department supervisor.

18.6 Logbooks must be reviewed quarterly by QA Staff.

18.7 The QA Staff performs periodic audits to evaluate compliance with this SOP.

19) Method Performance

19.1 Initial Demonstration of Proficiency- Each analyst must perform an initial demonstration of proficiency on a method and matrix basis with a successful analysis of four LCS where acceptable precision and accuracy are generated. The accuracy component must fall within LCS criteria. The precision component must be less than 20% for duplicate RPD data.

19.2 Method Detection Limits (MDLs) must be determined on an annual basis (at minimum) or whenever major modifications are performed.

19.3 Performance evaluation samples are processed periodically to assess method performance.

20) Summary of Changes

Table 20.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R06	9/1/13	CES	Reformatting. Changes to Calibration Procedure.
R07	10/1/14	CES	Addition of replicate readings criteria.
R08	9/15/16	CES	Updated document review and record retention criteria.
R08	9/15/16	CES	Section 11.2, 11.4, 11.5 – revised for the digestion of the calibration curve and ICV/CCV standards.

21) References and Related Documents

21.1 U.S. Environmental Protection Agency, "Method 7471A, Mercury in Solid or Semi-Solid Waste (Manual Cold-Vapor Technique)", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update III, June 1997.

21.2 U.S. Environmental Protection Agency, "Method 7471B, Mercury in Solid or Semi-Solid Waste (Manual Cold-Vapor Technique)", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IV, February 2007.

21.3 Cetac M6100A Mercury Analyzer Operators Manual, March 1997, Version 1.2

21.4 ALS Environmental Quality Assurance Manual, Revision (most current)

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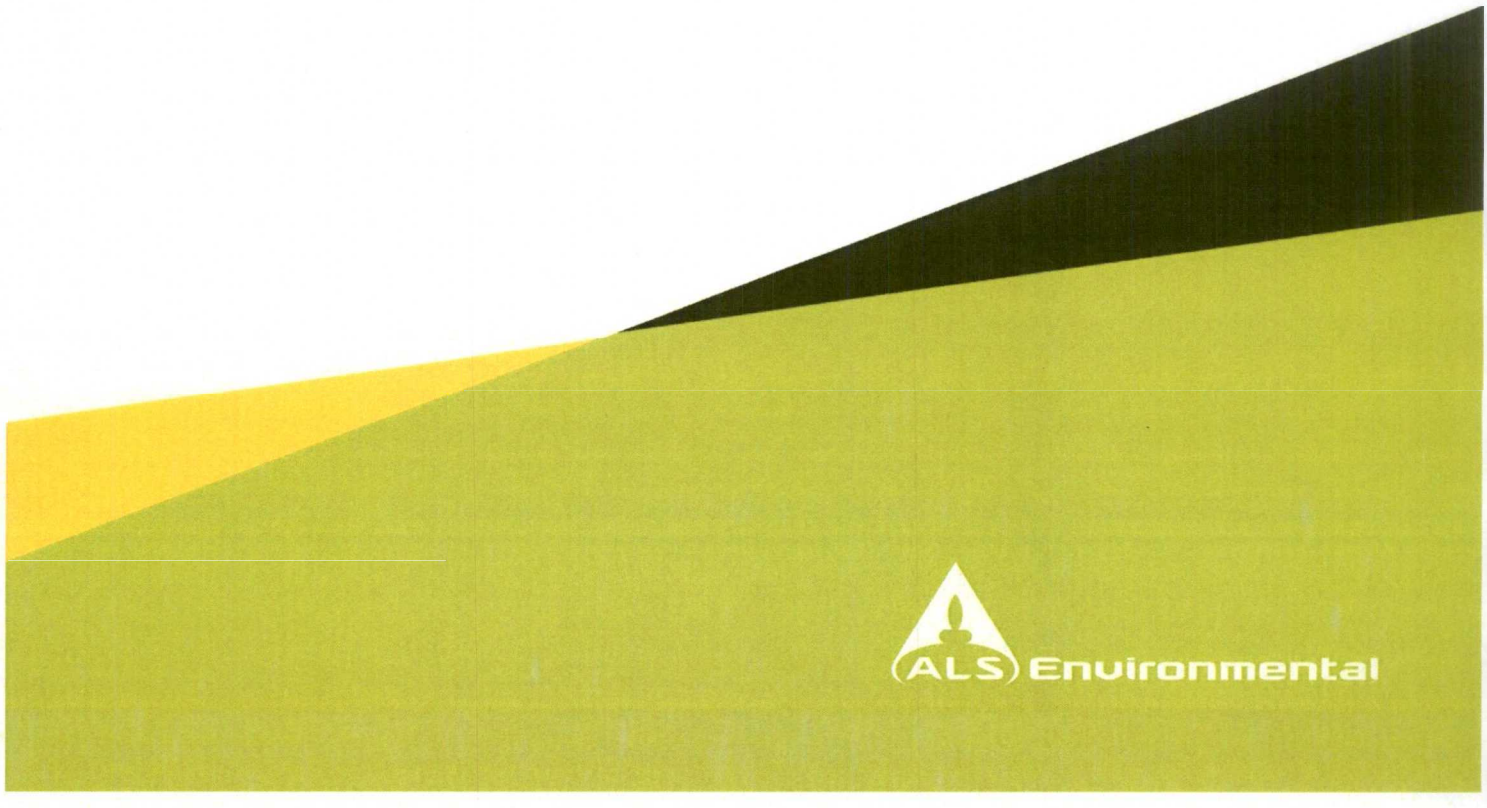
ALS Standard Operating Procedure

DOCUMENT TITLE:	PURGE & TRAP - SOLIDS
REFERENCED METHOD:	SW846 5035A
SOP ID:	HN-PT-004
REV. NUMBER:	R03
EFFECTIVE DATE:	04/01/2017



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PURGE & TRAP - SOLIDS

SW846 5035A

SOPID:	HN-PT-004	Rev. Number:	R03	Effective Date:	04/01/2017

Approved By: _____ Date: 3/7/17
Department Supervisor

Approved By: _____ Date: 3/7/17
Operations Manager

Approved By: _____ Date: 3/7/17
QA Manager

Approved By: _____ Date: 3/7/17
Laboratory Director

Archival Date: _____ Doc Control ID#: _____ Editor: _____

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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TABLE OF CONTENTS

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PURGE & TRAP - SOLIDS

1) Scope and Applicability

1.1 This SOP describes purge-and-trap procedures for the analysis of volatile organic compounds (VOCs) in solid samples (soils, sediments, and solid waste).

1.2 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.3 This SOP references and is based upon SW846 Method 5035A.

2) Summary of Procedure

2.1 Low Level Soil

2.1.1 A sample aliquot is transferred to a sealed container. At time of analysis, required reagents are added without breaking the seal, and an inert gas is passed through the sample with heating and agitation.

2.1.2 The vapor is swept through a sorbent column where the volatile components are adsorbed.

2.1.3 After purging is completed, the sorbent column is heated and back flushed with inert gas to desorb the components onto a gas chromatographic column.

2.2 Medium-High Concentration Soil Extracts

2.2.1 An aliquot of sample is combined with a known volume of methanol.

2.2.2 A portion of the methanol is combined with reagent grade water and purged according to the volatiles analytical SOP.

3) Definitions

3.1 Method Blank: A clean matrix to which all reagents are added in the same volumes or proportions as used in sample processing and carried through the complete analytical procedure.

3.2 Laboratory Control Sample (LCS): A clean matrix spiked with compound(s) representative of the target analytes.

3.3 Matrix: The component or substrate (e.g., surface water, groundwater, soil) that contains the analyte of interest.

3.4 Matrix Spike (MS/MSD): An aliquot of sample spiked with a known concentration of target analyte(s).

3.5 Stock Standard Solution: A concentrated solution containing certified standards that are the target or method analytes.

3.6 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples

4) Health and Safety Warnings

4.1 Lab Safety

4.1.1 Due to various hazards in the laboratory, safety glasses, disposable gloves, and laboratory coats or aprons must be worn when working with unknown samples. In addition, heavy-duty gloves and a face shield are recommended when dealing with toxic, caustic, and/or flammable chemicals.

4.1.2 The toxicity or carcinogenicity of each reagent used has not been precisely defined. However, each chemical used must be treated as a potential health hazard and exposure reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.2 Waste Disposal

4.2.1 Procedures for sample disposal are documented in SOP HN-SAF-001, *Waste Disposal Procedures.*

4.2.2 Samples must be disposed according to Federal, State, and local regulations.

4.3 Pollution Prevention

4.3.1 The quantities of chemicals purchased, when possible, must be based on the expected usage during its shelf life.

4.3.2 Standards and reagents must be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 Chloromethane may be lost if the purge flow is too fast.

5.2 Bromoform may be lost if the purge flow is too slow or the transfer line has cold spots/active sites.

5.3 Tetrachloroethane and 1,1-dichloroethane may deteriorate due to contamination and/or active sites in the purge and trap system.

6) Interferences

6.1 Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided. These compounds will result in interferences or false positives in the determinative step.

6.2 Methyl acetate and ethyl acetate artifacts may be formed with the addition of methanol in medium-high level soils. Additionally, an artifact is sometimes observed for acetone in the acidification of certain soils with sodium bisulfate.

6.3 At high desorption temperatures thermal decomposition products such as chloromethane, bromomethane, and iodomethane can be formed due to degradation or contamination on the analytical trap, especially with the addition of methanol in medium-high level soils. When levels of these compounds rise above the target detection limits, the analytical trap on the concentrator should be replaced and the

instrument should be recalibrated to ensure that the trap was not a source of contamination.

6.4 Carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of organic-free reagent water to check for cross-contamination. If a sample saturates the system and the sample immediately following has hits for the same compound(s) that saturated the system at a level ≤ 5xPQL, the second sample must be reanalyzed to determine if carryover occurred.

6.5 Special precautions must be taken to determine methylene chloride. The analytical and sample storage areas should be isolated from all atmospheric sources of methylene chloride. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel.

7) Personnel Qualifications and Responsibilities

7.1 This method must be used by, or under the supervision of, analysts experienced in volatile analysis by P&T techniques.

7.2 Analyst - It is the responsibility of the analyst(s) to:

7.2.1 Read, understand, and follow the method SOP.
7.2.2 Produce contractually compliant data that meets all quality requirements using this procedure
7.2.3 Complete the required demonstration of proficiency prior to working without supervision.

7.3 Department Supervisor - It is the responsibility of the department supervisor to:

7.3.1 Ensure analysts have the technical ability and training required for performance of this procedure.
7.3.2 Ensure analysts have completed the required demonstration of proficiency before performing this procedure without supervision.
7.3.3 Produce contractually compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

8) Sample Collection, Handling, and Preservation

8.1 All samples must be iced or refrigerated at 4° ± 2° C from the time of collection until analysis or freezing. Refer to Table 8.1 for sample containers, sample preservation, and sample holding time information.

Table 8.1 Volatile Organics Sample Collection, Preservation and Hold Time

Sample Matrix	Container	Preservative	Holding Time
Low-Level Soils (VOCs < 200ppb) by SW846-5035A	Collect with approved coring device (EnCore, etc) each ~5 gram sample in pre-tared 40 mL VOA vials, containing 5mL of organic free water, 1g sodium bisulfate & stir bar	4°C; Sodium bisulfate (or Sodium Triphosphate for Missouri samples)	48 hrs to transfer contents of core device to a preserved 40 mL VOA vial; analyze within 14 days from collection.

Table 8.1 Volatile Organics Sample Collection, Preservation and Hold Time			
Sample Matrix	Container	Preservative	Holding Time
Low-Level Soils (VOCs < 200ppb) by SW846-5035	Collect with approved coring device (EnCore, etc) each ~5 gram sample in pre- tared 40 mL VOA vials	4°C during transport then freeze at < -7°C but not < -20°C	48 hrs from collection to freezing process. Analyze within 14 days of collection.
Low-Level Soils (VOCs < 200ppb) by SW846-5035	Collect with approved coring device (EnCore, etc) each ~5 gram sample in pre- tared 40 mL VOA vials, containing 5 mL of organic free water & stir bar	4°C during transport then freeze at < -7°C but not < -20°C	48 hrs from collection to freezing process. Analyze within 14 days of collection
Low-Level Soils (VOCs < 200ppb) by SW846-5035	Collect with approved coring device (EnCore, etc) each ~5 gram sample in pre- tared 40 mL VOA vials containing a stir bar	4°C	Analyze within 48 hours of sample collection
Medium/High-Level Soils (VOCs >200 ppb) by SW846-5035	Collect with approved coring device (EnCore, etc) or field preserve samples in pre- tared 40 mL vials with methanol	4°C; Methanol	48 hrs to transfer contents of core device to a 40 mL VOA vial, containing 10 mL methanol; Analyze methanol extract within 14 days of collection

9) Equipment and Supplies

9.1 Syringe valve: Two-way, with Luer ends (three each), if applicable to the purging device.

9.2 Syringe: 10 mL, 5 mL, 1 mL, 1 µL, 10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL gas-tight

9.3 Balances: Analytical, capable of accurately weighing 0.0001 grams, top-loading capable of weighing 0.01 grams.

9.4 Pre- tared VOA vials: 40-mL, with screw cap, Teflon™ septa, and 10 mL P&T grade methanol

9.5 Volumetric flasks: 5 mL, 10 mL, 50 mL, and 100 mL class A with ground glass stoppers.

9.6 Disposable Pasteur pipettes (2 mL)

9.7 Auto-sampler vials with Teflon septa and screw top

9.8 Heater: Capable of maintaining the purging chamber to within ± 1°C over the temperature range of ambient to 40°C.

9.9 Atomx or Evolution Auto-sampler with sample heater and stirring mechanism

9.10 Purge and trap device: The purge and trap consists of a sample purging device and a trap.

9.10.1 Purge device: The recommended purging chamber is designed to accept 5 or 10 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as fine bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column.

9.10.2 Traps: Supelco K (VOACARB 3000), Teledyne #9 (proprietary), or equivalent.

10) Standards and Reagents

10.1 Methanol (MeOH) - Purge and trap grade (or equivalent).

10.2 Spiking Solutions

10.2.1 VOA Internal Standard/Surrogate Spiking Solution @ 20 ug/mL
10.2.2 VOA LCS/MS Spiking Solution @ 20 ug/mL
10.2.3 GRO Surrogate Spiking Solution @ 100 ug/mL
10.2.4 GRO LCS/MS Spiking Solution @ 20,000 ug/mL

10.3 For detailed information regarding the surrogate and/or analyte spike solutions, refer to the appropriate analytical SOP.

11) Method Calibration

11.1 Perform support equipment (balances, etc.) calibration checks as required for daily use.

12) Sample Preparation/Analysis

12.1 System Parameters:

12.1.1 Purge and Trap Concentrator - Recommended parameters:

Purge Conditions		
	Atomx	Evolution
Purge Flow	40 mL/min	40 mL/min
Purge	11 min. Ambient-35 °C (Low Level Soils-40°C)	11 min. Ambient-35 °C (Low Level Soils-40°C)
Desorb	0.75-2 min. @ 250°C	0.5 min. @ 260°C
Bake	5 min. @ 260°C	8 min. @ 255-265°C

12.1.2 The same purge and trap conditions must be used for purging of all standard, QC, and field samples.

12.2 Sample preparation

12.2.1 Laboratory QC Samples

12.2.1.1 Method Blank

12.2.1.1.1 A method blank must be purged with each analytical batch of ≤ 20 field samples processed during a 12 hour operating window (for low level soils) or with each extraction batch of≤ 20 field samples prepared in 8 hours (medium-high level soils).

12.2.1.1.2 Preparation:

12.2.1.1.2.1 Low Level Soil

12.2.1.1.2.1.1 Add 5 g of clean sand to a 40 mL VOA vial with a stirbar. Add the appropriate amount of preservative (see Table 8.1 and Section 12.1.2).

12.2.1.1.2.1.2 Cap and place in the auto-sampler.

12.2.1.1.2.2 Medium/High Level Soil (Methanol Dilution)

12.2.1.1.2.2.1 Add 10 g of clean sand to 10 mL of methanol

12.2.1.1.2.2.2 Sonicate for 20 minutes.

12.2.1.1.2.2.3 Add ~ 45 mL of DI water to a 50 mL volumetric flask.

12.2.1.1.2.2.4 Add 1000 uL of the methanol extract and bring to final volume with DI.

12.2.1.1.2.2.5 Transfer to a 40 mL VOA vial (no headspace), cap and purge.

12.2.1.2 Laboratory Control (LCS)

12.2.1.2.1 The LCS must contain the analytes of interest at or below the mid-point of the calibration curve.

12.2.1.2.2 The LCS must be analyzed with each analytical batch of ≤ 20 field samples processed during a 12 hour operating window (for low level soils) or prepared with each extraction batch of ≤ 20 field samples prepared in 8 hours (medium-high level soils).

12.2.1.2.3 Preparation

12.2.1.2.3.1 Low Level Soil

12.2.1.2.3.1.1 Add ~ 45 mL of DI water to a 50 mL volumetric flask.
12.2.1.2.3.1.2 Add 50 uL of the VOA LCS/MS Spiking Solution (Section 10.2.2) and bring to final volume with DI
12.2.1.2.3.1.3 Add 5 g of Ottawa sand to a 40 mL VOA vial with a stirbar.
12.2.1.2.3.1.4 Add 5 mL of diluted LCS/MS mix (12.2.1.2.3.1.2) to the vial and tightly cap.

12.2.1.2.3.2 Medium Level Soil (Methanol Dilution)

12.2.1.2.3.2.1 Add 10 g of Ottawa sand and 10 mL of methanol to a 40 mL VOA vial.
12.2.1.2.3.2.2 Sonicate for 20 minutes.
12.2.1.2.3.2.3 Add ~ 45 mL of DI water to a 50 mL volumetric flask.
12.2.1.2.3.2.4 Add 1000 uL of the methanol extract and 50 uL of the VOA LCS/MS Spiking Solution (Section 10.2.2) or 10 uL of the GRO LCS/MS spiking solution (Section 10.2.4) and bring to final volume with DI
12.2.1.2.3.2.5 Transfer to a 40 mL VOA vial (no headspace), cap.

12.2.1.3 Matrix Spikes (MS/MSD)

12.2.1.3.1 A MS/MSD pair must be purged with each analytical batch of ≤ 20 field samples processed during a 12 hour operating window (for low level soils) or with each extraction batch of≤ 20 field samples prepared in 8 hours (medium-high level soils).

12.2.1.3.2 Preparation

12.2.1.3.2.1 Low Level Soil

12.2.1.3.2.1.1 Using a gas tight syringe, add 5 uL of the VOA LCS/MS spiking solution (Section 10.2.2) directly to the 40 mL VOA vial specified for MS/MSD use. The spike must be added through the septum. ***Do not uncap the VOA vial.***

12.2.1.3.2.2 Medium Level Soil (Methanol Dilution)

12.2.1.3.2.2.1 Sonicate the sample for 20 minutes.
12.2.1.3.2.2.2 Add ~ 45 mL of DI water to a 50 mL volumetric flask.

12.2.1.3.2.2.3 Add 1000 uL of the methanol extract and 50 uL of the VOA LCS/MS Spiking Solution (Section 10.2.2) or 10 uL of the GRO LCS/MS spiking solution (Section 10.2.4) and bring to final volume

12.2.1.3.2.2.4 Transfer to a 40 mL VOA vial (no headspace), cap.

12.3 Field Samples

12.3.1 Low Level Soil

12.3.1.1 Allow samples to warm to room temperature.
12.3.1.2 Place in the Atomx/Evolution auto-sampler and create the appropriate sequence.
12.3.1.3 The auto-sampler automatically adds water, internal standard/surrogate solution, and with heating/stirring purges the sample.

12.3.2 Methanol Dilution (Medium-High Level Soil) Samples

12.3.2.1 Weigh the pre-tared VOA vial to determine the mass of sample provided.
12.3.2.2 If necessary, add sufficient methanol to maintain a 1:1 ratio (i.e., 10 g of sample to 10 mL of methanol).
12.3.2.3 Sonicate for 20 minutes.
12.3.2.4 Add ~ 45 mL of DI water to a 50 mL volumetric flask.
12.3.2.5 Add 1000 uL of the methanol extract and bring to final volume
12.3.2.6 Transfer to a 40 mL VOA vial (no headspace), cap and purge.

13) Troubleshooting

13.1 N/A

14) Data Acquisition

14.1 N/A

15) Calculation, and Data Reduction Requirements

15.1 N/A

16) Quality Control, Data Assessment and Corrective Action

16.1 Method Blank

16.1.1 See determinative method SOP for frequency, acceptance criteria, and corrective action.

16.2 Laboratory Control Samples (LCS)

16.2.1 See determinative method SOP for frequency, acceptance criteria, and

corrective action.

16.3 Matrix Spike

16.3.1 See determinative method SOP for frequency, acceptance criteria, and corrective action.

16.4 Surrogates

16.4.1 See determinative method SOP for frequency, acceptance criteria, and corrective action.

17) Data Records Management

17.1 All records must be maintained for a period of no less than 10 years.

17.2 Document each sample prep batch into the LIMS prep batch.

17.3 All anomalies noted during the preparative process must be documented in the LIMS prep batch.

17.4 All reagents and chemicals purchased and/or prepared must be labeled, entered into LIMS and traceable back to the respective chemical inventory and reagent/standard preparation logbooks.

18) Quality Assurance and Quality Control

18.1 Logbooks must be reviewed monthly by the department supervisor.

18.2 Logbooks must be reviewed quarterly by the QA Staff.

18.3 The QA Staff must conduct periodic audits to evaluate compliance with this SOP.

19) Contingencies for Handling Out of Control Data

19.1 Ideally, data should never be reported when the associated QC data fail criteria. All samples must be re-run if possible.

19.2 Should sample re-analysis not be possible, the data can only be reported with the use of appropriate data qualifiers and project narration.

19.3 Deviations from this procedure or the quality control criteria outlined within must be documented via the NC/CA database for further evaluation.

20) Method Performance

20.1 Refer to the determinative method for performance data.

20.2 MDL's are performed annually, at a minimum and whenever major modifications to this procedure occur.

21) Summary of Changes

Table 21.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R02	8/1/12	CES	Formatting
R02	8/1/12	AK	Updating

R02	8/1/12	CDW	Updating
R03	4/1/17	CES	Update document review and record retention criteria.
R03	4/1/17	CES	Updated auto-sampler IDs and purge conditions (Section 12.1.1)

22) References and Related Documents

22.1 ALS Environmental Quality Assurance Manual, Revision (most current)

22.2 U.S. Environmental Protection Agency, "Method 5035A Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples", Revision 1, July 2002.

22.3 VOA Spike Volumes (Table 22.3)

22.4 GRO Spike Volumes (Table 22.4)

Table 22.3 VOA Spike Volumes

Parameter	*Internal Standard/Surrogate Addition*	*Spike Addition*
VOA Method Blank	5 uL @ 20 ug/mL by Autosampler	----------
VOA LCS	5 uL @ 20 ug/mL by Autosampler	50 uL @ 20 ug/mL to 50 mL water and 1 mL methanol.
VOA MS/MSD	5 uL @ 20 ug/mL by Autosampler	50 uL @ 20 ug/mL to 50 mL water and 1 mL sample
VOA Samples	5 uL @ 20 ug/mL by Autosampler	----------

Table 22.4 GRO Spike Volumes

Parameter	*Surrogate Addition*	*Spike Addition*
GRO Method Blank	5 uL @ 100 ug/mL by Autosampler	----------
GRO LCS	5 uL @ 100 ug/mL by Autosampler	10 uL @ 20,000 ug/mL to 50 mL water and 1 mL methanol.
GRO MS/MSD	5 uL @ 100 ug/mL by Autosampler	10 uL @ 20,000 ug/mL to 50 mL water and 1 mL sample
GRO Samples	5 uL @ 100 ug/mL by Autosampler	----------

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ALS Standard Operating Procedure

DOCUMENT TITLE:	SEMI-VOLATILE ORGANIC COMPOUNDS
REFERENCED METHOD:	SW846 8270C / 8270D / EPA 625
SOP ID:	HN-SMS-001
REV. NUMBER:	R08
EFFECTIVE DATE:	09/15/2016

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SEMI-VOLATILE ORGANIC COMPOUNDS

SW846 8270C / 8270D / EPA 625

SOPID:	HN-SMS-001	Rev. Number:	R08	Effective Date:	09/15/2016

Approved By: _____ Date: 8/25/16
Department Supervisor

Approved By: _____ Date: 8/29/16
Operations Manager

Approved By: _____ Date: 8/25/16
QA Manager

Approved By: _____ Date: 8/26/16
Laboratory Director

Archival Date: _____ Doc Control ID#: _____ Editor: _____

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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TABLE OF CONTENTS

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SEMI-VOLATILE ORGANIC COMPOUNDS

1) Scope and Applicability

1.1 This method can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols.

1.2 This method, and associated extraction procedures, is applicable to a variety of matrices including: drinking water, non-potable water, solid/chemical materials, biological tissues, and air/emissions. This method is based upon and compliant with SW846 8270C, 8270D, and EPA 625.

2) Summary of Procedure

2.1 Samples are extracted utilizing the appropriate method/SOP for semivolatile extraction of the sample matrix type. The extracts are analyzed using GC/MS techniques. The routinely reported standard target compound list are those compounds marked with an * in Table 2.1.

2.2 Typical Method Quantitation/Reporting Limit (MQL/MRL) for this method for determining an individual compound is approximately 33 ug/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes and approximately 5 ug/L for groundwater samples. MQL/MRLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector or chromatographic system. MQL/MRLs, and all other calculated concentrations, shall be based upon values obtained from sample extracts processed according to this SOP and the applicable extraction procedure SOP HN-EXT-001, HN-EXT-002, HN-EXT-003, HN-EXT-013, or HN-EXT-016.

TABLE 2.1 -- CHARACTERISTIC IONS FOR SEMI-VOLATILE COMPOUNDS[2]

Compound	Primary Ion	Secondary Ion(s)	Water MQL[1] µg/L	Solid MQL[1] µg/Kg
1,1'-Biphenyl*	154	153, 152, 76	1	33
1,2,4,5-Tetrachlorobenzene*	216	214, 218	5	333
1,2,4-Trichlorobenzene	180	182, 145	1	33
1,2-Dichlorobenzene	146	148, 111	1	33
1,2-Dinitrobenzene	168	50, 63	1	33
1,2-Diphenylhydrazine (azobenzene)	77	105, 182	1	33
1,3-Dichlorobenzene	146	148, 111	1	33
1,3-Dinitrobenzene	168	76, 75, 122	1	67
1,4-Dichlorobenzene	146	148, 111	1	33
1,4-Dichlorobenzene-d_4 (I.S.)*	150	152, 115	1	33

TABLE 2.1 -- CHARACTERISTIC IONS FOR SEMI-VOLATILE COMPOUNDS[2]				
Compound	**Primary Ion**	**Secondary Ion(s)**	**Water MQL[1] µg/L**	**Solid MQL[1] µg/Kg**
1,4-Dinitrobenzene	168	75, 76	1	33
1,4-Dioxane*	88	58, 43	5	167
1,4-Naphthoquinone	158	102, 130	5	167
1-Napthylamine	143	115, 116	5	667
2,2'-Oxybis(1-Chloropropane)*	45	77, 121	1	33
2,3,4,6-Tetrachlorophenol*	232	131, 230	1	67
2,4,5-Trichlorophenol*	196	198, 132	1	33
2,4,6-Tribromophenol (surr.)*	330	332, 141	1	33
2,4,6-Trichlorophenol*	196	198, 132	1	33
2,4-Dichlorophenol*	162	164, 98	1	33
2,4-Dimethylphenol*	107	122, 121	1	33
2,4-Dinitrophenol*	184	91, 107	5	33
2,4-Dinitrotoluene*	165	63, 89	1	33
2,6-Dichlorophenol	162	164, 98	1	33
2,6-Dinitrotoluene*	165	63, 89	1	33
2-Acetylaminofluorene	181	180, 223	5	167
2-Chloronaphthalene*	162	127, 164	0.1	6.67
2-Chlorophenol*	128	64, 130	1	33
2-Fluorobiphenyl (surr.)*	172	171	1	33
2-Fluorophenol (surr.)*	112	64	1	33
2-Methylnapthalene*	142	141, 115	0.1	6.67
2-Methylphenol*	107	108, 77	1	33
2-Naphthylamine	143	115, 116	5	667
2-Nitroaniline	65	92, 138	1	33
2-Nitrophenol	139	109, 65	1	33
2-Picoline	93	66, 92	5	330
3,3'-Dichlorobenzidine*	252	254, 126	5	167
3,3'-Dimethylbenzidine	212	213, 211	25	667
3&4-Methylphenol*	107	108, 77	1	33

TABLE 2.1 -- CHARACTERISTIC IONS FOR SEMI-VOLATILE COMPOUNDS[2]				
Compound	**Primary Ion**	**Secondary Ion(s)**	**Water MQL[1] µg/L**	**Solid MQL[1] µg/Kg**
3-Nitroaniline*	138	65, 92	1	33
4,6-Dinitro-2-methylphenol*	198	51, 105	1	33
4-Aminobiphenyl	169	168, 170	5	667
4-Bromophenyl phenyl ether*	248	250, 141	1	33
4-Chloro-3-methylphenol*	107	144, 142	1	33
4-Chloroaniline*	127	129, 92	1	67
4-Chlorophenyl phenyl ether*	204	206, 141	1	33
4-Nitroaniline*	138	108, 92	1	167
4-Nitrophenol*	65	109, 139	5	33
4-Terphenyl-d14 (surr.)*	244	122, 212	1	33
5-Nitro-o-toluidine	152	77, 106	5	167
7,12-Dimethylbenz(a)anthracene	256	241, 239	1	167
a,a-Dimethylphenylamine	58	91, 42	5	167
Acenaphthene*	154	153, 152	0.1	6.67
Acenaphthene-d^{10} (I.S.)*	164	162, 160	1	33
Acenaphthylene*	152	151, 153	0.1	6.67
Acetophenone*	105	71, 51, 120	1	33
Aniline	93	66, 65	1	33
Anthracene*	178	176, 179	0.1	6.67
Atrazine*	200	215, 68, 173	1	33
Benzaldehyde*	77	106, 105, 51	1	67
Benzidine	184	92, 185	5	667
Benzo(a)anthracene*	228	229, 113	0.1	6.67
Benzo(a)pyrene*	252	253, 125	0.1	6.67
Benzo(b)fluoranthene*	252	253, 125	0.1	6.67
Benzo(g,h,i)perylene*	276	138, 277	0.1	6.67
Benzo(k)fluoranthene*	252	253, 125	0.1	6.67
Benzoic acid	122	105, 77	20	167
Benzyl Alcohol	108	79, 77	1	33

TABLE 2.1 -- CHARACTERISTIC IONS FOR SEMI-VOLATILE COMPOUNDS[2]				
Compound	**Primary Ion**	**Secondary Ion(s)**	**Water MQL[1] µg/L**	**Solid MQL[1] µg/Kg**
Bis(2-chloroethoxy) methane*	93	95, 123	1	33
Bis(2-chloroethyl) ether*	93	63, 95	1	33
Bis(2-ethylhexyl)phthalate*	149	167, 279	1	33
Butyl benzyl phthalate*	149	91, 206	1	33
Caprolactam*	55	113, 56, 42	5	33
Carbazole*	167	139, 83	1	33
Chrysene*	228	226, 114	0.1	6.67
Chrysene-d_{12} (I.S.)*	240	120, 236	1	33
Dibenzo(a,h)anthracene*	278	139, 279	0.1	6.67
Dibenzo(a,h)acridine	279	280, 277, 250	5	67
Dibenzofuran*	168	139, 84	1	33
Diethylphthalate*	149	177, 150	1	33
Dimethylphthalate*	163	194, 164	1	33
Di-n-butylphthalate*	149	150, 104	1	33
Di-n-octylphthalate*	149	150, 279	1	33
Diphenylamine	169	168, 167	1	33
Ethyl methanesulfonate	79	109, 97	5	167
Fluoranthene*	202	101, 203	0.1	6.67
Fluorene*	166	165, 167	0.1	6.67
Hexachlorobenzene*	284	142, 249	1	33
Hexachlorobutadiene*	225	223, 190	1	33
Hexachlorocyclopentadiene*	237	235, 272	5	33
Hexachloroethane*	117	201, 199	1	33
Hexachlorophene	196	198, 209, 211, 406, 408	80	1670
Hexachloropropene	213	211, 215	5	167
Indeno(1,2,3-cd)pyrene*	276	138, 227	0.1	6.67
Isophorone*	82	95, 138	5	167
Isosafrole	162	131, 161	5	333
Methylmethanesulfonate	80	79, 65	5	333